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To: . Mr. A. I. Palmer

Date: February 14, 1978

From: . G. D. Keritsis

Subject: . PM Case #641. Addendum to PM Case No. 653

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As you are aware, the latest draft patent application for PM Case #641 has combined the PM Cases 653, 689, 698, 713, 742 and sub-portion of 641 covering the foaming/expansion of smoking materials.

The major problem with this draft, as I see it, is that certain technologies such as those of PM Case Nos. 653, 689, 742 and the foaming/expansion section of 641 are also applicable to tobacco. Since PM Case #641 does not cover this very important utilization area, I feel a separate case should be prepared to especially cover the extrusion of various shaped tobacco articles such as the direct extrusion of tobacco cigarette rods, etc.

As you probably are aware, (see attached memo to Dr. W. F. Gannon from G. D. Keritsis titled, "Development Program-Extrusion of Cigarette Rods" and also the attached process schematic), such work of extruding tobacco articles is being going on for a long time and several demonstration samples have been produced. Also, the attached schematic and such articles as extruded porous (foamed) cigarette rods or hollow tobacco tubes with/without the use of cigarette wrapping paper were presented to Marketing and others by Mr. K. S. Burns and Dr. W. F. Gannon on November, 1977 and January, 1978. Such samples were shown to Dr. T. Laszlo and the use of microwave technology to dry and expand them was also discussed. Dr. T. Laszlo is very much enthused with this technology and very confident that the existing microwave set up used to produce the cellulose acetate wrappless cigarette filter will also work in this case to produce the desired products. The evaluation of this will now follow as soon as the unit becomes available.

I, as well as Mr. L. Meyer and Dr. T. Laszlo, feel that this type of technology will probably become the process of the future for making cigarettes and other desired shapes of smoking articles, and as such we would like a good patent protection.

This process will primarily involve the formation of a tobacco dough with water and a binder adhesive. Such a dough will contain at least 50% solids, usually 50-70%, and will then be extruded under pressure to ambient through a die of a desired shape with the use of a plastic extruder or pasta making type units to produce the desired extrudate. Such extrudates could be in the form of a porous (foamed) cigarette rod, a tubular (single or multiple) cigarette rod or star-like in cross section or other. Reconstituted tobaccos to be blended with other tobaccos or made into 100% cigarettes in the form of hollow shreds, noddles, etc., could also be made by this process. The extruded articles could be foamed/expanded either as the extrudate exits the die or afterwards as the extrudate is being dried

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with heat or microwave. Certain blowing agents such as $(\text{NH}_4)_2\text{CO}_3$, carbamate, etc., could be incorporated in the dough to expand the extrudate with gases that are being generated when these blowing agents are being decomposed with heat or acid or other means. To prevent the premature expansion of the dough in the confined extruder barrel or before the dough exits the die, a pressurized system and/or proper temperature conditions are used to prevent either the decomposition of the blowing agent or its premature gasification in the barrel. Then, the compressed/dissolved gases in the tobacco dough upon exiting of the extrudate through the die to a lower (ambient) pressure cause the extrudate to foam/expand, in many cases in the confinement of sizing dies. In the case that the blowing agent had not been gasified (decomposed) in the extruder, such a decomposition (gasification) could be accomplished within the post sizing dies in a post-expansion type treatment. Similarly, gases, liquid or dispersed blowing agents could be added under pressure in the last zone of the extruder to be mixed (dissolved) in the dough at an elevated temperature (above the boiling, gasification point of the liquid at ambient pressure). This pressure keeps the gaseous materials dispersed in the dough in a manner similar to carbonaceous soft drinks, and when the pressure is released upon exiting the extruder die, the gases, liquids, expand causing the extrudate to be foamed. With this technique the water which is used to make the dough or steam that could be added in the last extruder zone under pressure could be the blowing agent for these extrudates if the temperature of the last pressurized zone is above the boiling point of water. Similarly the extrudate water could also be used in post expanding it with heat or microwave within sizing dies during the drying process of the extrudate.

Much of this technology for foaming/expanding extrudates has been previously disclosed by the attached three memos to Mr. L. Meyer by G. Keritsis.

Below, I list a few examples that were carried out in the laboratory with improvised extrusion/shaping techniques to demonstrate the feasibility of such products.

Example 1

A dough-like slurry was made as follows:

Sixteen (16) grams of guar gum (General Mills) and 16 g of Na-Alginate were dry blended and then wetted with 67 g of water. To this gum dough 168 g of 40 mesh Marlboro filler dust was added together with an additional amount of 67 g of water. The formed dough was kneaded (worked) well to insure that the tobacco and the gums were well mixed and wetted. The tobacco-gum dough was then divided into two equal weight samples, Sample A and B. To the first sample, Sample A, 6 g of $(\text{NH}_4)_2\text{CO}_3$ was added and mixed therein. The compositions derived were:

| | A | B |
|------------------------------|------|------|
| Guar Gum | 8 g | 8 g |
| Na-Alginate | 8 g | 8 g |
| Tobacco dust | 84 g | 84 g |
| $(\text{NH}_4)_2\text{CO}_3$ | 6 g | - |
| Water | 67 g | 67 g |

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These doughs, A and B, were then processed to produce the samples described below:

a) A portion of A or B dough was rolled on a flat surface to produce various cylindrical rods ranging in circumference from about 20 to 100 mm. These cylindrical rods were then dried in 120°C oven. The dried rods were then removed from the oven, cut into 50 to 100 mm in lengths and examined by puffing and a microscope. The results indicate that the rods made with Sample A-dough were porous (foamed) and easy to draw, whereas the rods of Sample B were not as porous and very hard to draw. On smoking, Sample A produced a mild smoking material which was burning statically. Sample B did not burn as well and didn't produce much smoke.

b) Similar cylindrical rods as for the previous example were made but prior to drying, the rods were immersed in a 25% CaCl_2 and 0.5% HCl aqueous solution for 20 seconds in an attempt to set (cross-link) the alginic acid with Ca^{++} and possible foam the formulation A by reacting the $(\text{NH}_4)_2\text{CO}_3$ with the acid (HCl). Parts of the treated rods were air dried, whereas the remaining portions were dried in 125°C oven. The results indicate that all rods (A or B) were moisture (water) insensitive and rigid. Sample A rods were porous and easy to draw. The higher porosity was found to be with the Sample A that was dried in the oven.

c) Similar cylindrical rods as for previous examples were made with the difference being that the tobacco doughs were rolled around a glass rod of 15 mm in diameter. The glass rod was then removed from the center of the cylindrical doughs. The produced hollow cylindrical tobacco rods (tubes) were then treated as for "a or b" steps of this example 1. The tubes did not collapse during these treatments and when made into filtered cigarettes with/without the use of a cigarette paper wrapper were smoked. The tubes burned satisfactorily.

d) Similar rods as for "a" step were made. With the use of a stainless steel wire multiple channels were introduced along the cylindrical cross sections. These rods were then treated as per "a or b" steps and were found to produce satisfactory burning and smoking products.

e) Finally, a portion of the doughs, A or B, was made into 25 mm in circumference cigarette rods with the use of a RYO filtermatic cigarette maker supplied by the Suttcliff Tobacco Co. of Richmond, Va. with/without the use of a cigarette wrapping paper. The rods were then dried and tested as per step "a" of this example with similar observations.

Example 2

Similar doughs as per example 1 were made and extruded into various shaped articles such as noodles, rods, crosses, hollow tubes using a Torchio pasta maker. The extrudates were then treated as per example 1 producing similar results.

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Example 3

Various doughs were made with 80 and 90 parts RCB-tobacco dust (about 40 mesh), 20 and 10 parts, respectively, of either guar, CMC, xanthan, methocel, etc. gums. Enough water was then used to produce doughs having 60% solids and 40% water. To part of the doughs ammonium carbonate was added to introduce about 5% $(\text{NH}_4)_2\text{CO}_3$ on the weight of the dough. These doughs were then extruded into filament (shred) like products with the use of single circular hole die attached at the end of a barrel (cylinder) which was equipped with a proper plunger (piston) (Fisher Scientific Cat. No. 11-504-200). The barrel was loaded with the particular dough and pushed with the pressurized piston out of the die. The produced filaments resembling spaghetti were then dried as per example 1. The best results in regards to strength were achieved with the 20% gum products. The materials that contained the Xanthan were found to be the most flexible.

Example 4

Similar formulations as per examples 1 and 3 were made and processed into various shaped articles with the use of a play dough shaping machine. The results were similar as those previously noted.

Example 5

Tobacco dust, 60 g, was wetted with 40 g water. This wet tobacco was then placed in a hydraulic press chamber and pressed. The tobacco strongly adhered to itself forming a solid "plug".

In conclusion it appears that acceptable extruded, molded, tobacco products can be made with the incorporation of a gum adhesive (at least 10%), a solvent (water or other), a blowing agent (in many cases the solvent could be the blowing agent), heat (microwave) and pressure to produce unique, novel products that could be recognizably different in appearance or performance (subjectives and smoke deliveries).

Although a plastic extruder was not used to process such products, because of availability, the improvised techniques clearly indicate that this to be feasible and if anything more acceptable products could more easily be made with the proper equipment that we presently try to build or purchase.

In addition to the extrusion process, attempts are being made to transfer some of the cross-linking/stiffening studies from the laboratory to production, especially to treating RL-reconstituted tobacco to increase its filling capacity. This technology is covered in PM Case #689. This case, I also feel, should be patented for treating tobacco, and it should be compared with that of the A. T. Lendvay case for treating tobacco with aluminum sulfate for possible conflicts.

/jhb

cc: K. S. Burns (w/o attach)
W. F. Gannon (w/o attach)
✓ R. B. Seligman (w/o attach)

S. D. Keittis

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